Guideline 62-1 PRELIMINARY ANALYSIS OF PRODUCT SAMPLES

1. Description of analytical methods for each active ingredient.

ANALYTICAL METHOD

ANALYSIS OF DECANOIC, NONANOIC, AND OCTANOIC ACID IN ACID SANITIZER

APPARATUS: Liquid Chromatograph
Refractive Index Detector (Waters 410 or equivalent)
Waters NOVA-PAC C18 Radial Compression Cartridge (or equivalent)

SCOPE: This method may be used for the analysis of decanoic, nonanoic, and octanoic acid in acid sanitizer products. The procedure is especially useful for analysis of F-1592 ans F-1593.

COLUMN: Waters NOVA-PAC C18 Radial Compression Cartridge with a C18 Guard column.

DETECTOR: Refractive Index Detector set at 7.8×10^{-5} RIU full scale. Use the following settings for the Waters 410 Differential Refractomenter: SENS = 64 and SF = 1.

ELUENT: Acetonitrile:Water 50:50 V/V with 0.1% Acetic Acid

FLOW RATE: 1.0 mL/min

SAMPLE INJECTION SIZE: 0.02 mL for the unknown and standard solutions

UNKNOWN SAMPLE PREPARATION: Because of the unique solubility properties of the F-1592 and F-1593 acid sanitizers, it is best to inject these samples directly without any further dilution.

STANDARD SOLUTION: Accurately weigh out 0.27 - 0.33 gm of decanoic acid, 0.27 - 0.33 gm of nonanoic acid, and .008 - .012 gm of octanoic acid into a 10 mL volumetric flask. Each of these standard acids should have a minimum purity of 98%. Dilute this sample to volume with the eluent.

NOTE: The chromatographic conditions may be adjusted to suit the equipment being used.

QUANTITATION METHOD: Peak Area - The area may be determined using an electronic integrator or the area for each peak may be calculated by multiplying the height of the peak times the width of the peak at half height.

CALCULATIONS:

$$\% \text{ ANALYTE IN THE UNKNOWN SAMPLE} = \frac{(A_u)\ (C_s)\ (100)}{(A_s)\ (D_u)}$$

(A_u) = peak area of the unknown sample

(A_s) = peak area of the standard solution

(D_u) = gm/mL; Density of the unknown sample solution

(C_s) = gm/mL concentration of the analyte in the standard solution (Example: 0.3gm/10mL = 0.03gm/mL)

TABLE 1

ANALYTE	APPROXIMATE RETENTION TIME (MIN)
OCTANOIC	8 MIN
NONANOIC	12 MIN
DECANOIC	18 MIN

PREPARED BY: Chris Foor / 1/2/90 DATE

CHECKED BY: Glen L. Winiarski / 1/2/90 DATE

APPROVED BY: Murray Winiarski / 1/2/90 DATE

ANALYTICAL METHOD

DETERMINATION OF PHOSPHORIC ACID AND SULFURIC ACID IN ACID SANITIZER

SCOPE:

This procedure may be used for the analysis of phosphoric and sulfuric acids in F-1592, where propionic acid is present at 10 % ± 1 %, and pelargonic and decanoic acid are each present at levels of 3 % ± 0.3 %.

REAGENTS AND APPARATUS:

Buffer Solutions (4,7 or 10)
Deionized or Distilled Water
0.1 N Sodium Hydroxide Standard Solution
Analytical Balance
50 mL Buret
250 mL Beaker
Magnetic Stirrer and Magnetic Bar
pH Meter with Glass and Calomel Electrode

PROCEDURE:

Accurately weigh into a 250 mL beaker 1.00 gm of the sample to be analyzed. Dilute with deionized water to a total volume of about 100 mL. Titrate the sample with 0.1 N sodium hydroxide using a standardized pH meter to monitor the pH. Determine the volume of sodium hydroxide required to reach the first end point (V_1) which should occur around pH 3-4. Also determine the total volume of sodium hydroxide required to reach the second end point (V_2) which should occur around pH 8.5 - 9.5.

CALCULATIONS:

The end points V_1 and V_2 (mLs) should be determined by plotting a graph of the titration data. The concentration of decanoic, pelargonic, and propionic acids must be determined prior to this analysis using independent techniques. Their presence must be accounted for by subtracting the appropriate volume of sodium hydroxide from V_2 for each of these ingredients according to the following formula.

$$V_2 \text{ corrected} = V_{2C} = V_2 - \left[\frac{(\text{gms sample})\ 10}{\text{Normality NaOH}} \left(\frac{\% \text{ decanoic acid}}{172} + \frac{\% \text{ pelargonic acid}}{158} + \frac{\% \text{ propionic acid}}{74} \right) \right]$$

The concentrations of phosphoric and sulfuric acids are then calculated as follows:

$$\% \text{ (w/w) } H_3PO_4 = \frac{N \times (V_{2C} - V_1) \times .098 \times 100}{\text{gm of sample}}$$

$$\% \text{ (w/w) } H_2SO_4 = \frac{N \times (2V_1 - V_{2C}) \times .049 \times 100}{\text{gm of sample}}$$

Where N = normality of sodium hydroxide

Prepared by: _____ 1-17-90
Date

Checked by: _____ 1-17-90
Date

Approved by: _____ 1/17/90
Date

2. The following are results of the preliminary analysis of Econosan.

Active Ingredient/ Test Method	Time of Manufacture % w/w
Pelargonic Acid West No. 183	2.96
Decanoic Acid West No. 183	2.87
Phosphoric Acid West No. 191	8.52
Sulfuric Acid West No. 191	9.24

West Agro, Inc. ECONOSAN

Ref.: WAG-2A

Guideline 62-2 CERTIFICATION OF INGREDIENT LIMITS

CERTIFICATION STATEMENT

I hereby certify that, for purposes of FIFRA sec. 12(a)(1)(C), the description of the composition of ECONOSAN refers to the composition set forth on the Statement of Formula and supporting materials. This description includes the representations that: (1) no ingredient will be present in the product in an amount greater than the upper certified limit or in an amount less than the lower certified limit specified for that ingredient in a currently approved Statement of Formula; and (2) if the Agency requires that the source of supply of an ingredient be specified, that all quantities of such ingredient will be obtained from the source specified in the statement of formula.

Company Official: Ken J. Morris Date 9 February 1990
Director, Quality Assurance &
Government Regulatory Affairs

The Confidential Statement of Formula for Econosan states upper and lower certified limits of each ingredient. The formula is included in the Administrative Materials and a copy has been inserted into the confidential attachment to Volume 2 of this application.

Guideline 62-3 Analytical methods to verify certified limits

The analytical methods used to verify certified limits for the active ingredients are identical to the methods presented in Guideline 62-1, pages 4-7 of this study.

Analytical methods used to verify certified limits for inert ingredients immediately follow this page.